Batch Reactor Manual 5 –

Atlas and Globe 3 Bar Pressure Systems

Syrris Ltd

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1 Introduction

Thank you for purchasing an Atlas or Globe 3 Bar Pressure System from Syrris.

These instructions have been carefully prepared to guide the installer and end-user through the installation and use of the Atlas Potassium 3 Bar Pressure System and Globe 3 Bar Pressure System.

Before attempting to use this product, please read this manual thoroughly and follow the instructions carefully. In doing so, you will ensure the safety of yourself and others around you and you can look forward to your purchase giving you a long and satisfactory service.

Designed specifically for ease of use, all Atlas systems can be scaled in three ways:

- by volume
- automation level (manual, automated control from the Base Unit or full PC software control)
- number of reactions.

All of the Syrris systems have high chemical resistance.

This manual is designed for use in conjunction with Batch Reactor Manual 1 – Atlas Systems (PN:2000255) and Batch Reactor Manual 6 – Globe Systems (PN:2000260). These two manuals will provide all the information about the installation and operation of the Atlas and Globe systems. This manual is intended to highlight the use of the specific pressure parts incorporated into the system. Prior to using a Syrris pressure system, please read the corresponding manual thoroughly before reading this manual.

2 Safety

All Syrris reactors are designed and manufactured to ensure safe operation when used within their prescribed temperature and pressure limits, but the basic responsibility for safety using the equipment rests entirely with the user. This equipment should only be used by competent, suitably trained personnel after they have read and understood the operating instructions and considered any hazard involved.

2.1 Warning and Safety Information for Atlas Potassium 3 Bar Pressure Systems and Globe 3 Bar Pressure Systems

2.1.1 General Precautions

The system must only be pressurised when installed in a suitable protective fume hood with a closed shatterproof door.

Equipment should be prominently labelled when left pressurised or under vacuum. Use appropriate safety accessories and utilise appropriate Personal Protective Equipment (PPE).

2.1.2 Installation

Installation must be carried out by a Syrris Engineer or an appointed representative.

Place the equipment on a solid, horizontal, fire-proof surface. Ensure the area around the unit is clear.

Safety valves, burst discs and other methods of pressure release should be sited so as to minimise the chance of injury to people or damage to equipment if they should operate.

2.1.3 Operational Safety

Because of the high risk of injury or major damage which may occur with the failure of any part of a pressure system, it is essential that such systems are set up, used and maintained in a safe manner. Due to the brittle nature of glass, it is essential that safety screens are used and the system is installed in a suitable protective fumehood with a closed shatterproof door. Flying fragments from an exploding glass vessel can be fatal.

The user must have a complete understanding of the reaction planned to be run and carry out a risk assesment before starting the reaction. Only run reactions which have been assessed and ensure that the temperature and pressure variations are suitable for the chosen system. Risks to be evaluated include (but are not limited to): Use of hazardous and corrosive chemicals, rate of gas generation, possibility of thermal runaway, possibility of thermal decomposition of reactor content, effects of scale.

Comply with all safety and accident prevention regulations applicable to laboratory work. Operate in full compliance with local safety codes and rules.

The user must ensure that the reactor, safety manifold and all attached hardware will not be attacked by the chemical substances used.

Appropriate PPE must be worn at all times when operating the system, taking into account pressure, temperature and chemical hazards.

Before every use the reactor system must be examined by a competent person for any damage. The safety manifold must be visually checked for corrosion and blockages. Glass surfaces must be checked for scratches and chips. If any damage is observed the equipment MUST NOT be used. It should be disposed of or returned to Syrris for evalution.

Always ensure that the input pressure to the system is LESS than the maximum allowable working pressure for the system.

The user must ensure that there are adequate safety systems to prevent reactor pressure exceeding maximum allowable working pressure as displayed on the reactor.

Do not overfill the vessel and account for any thermal expansion during a reaction.

The pressure relief valve is factory set to open at 2.75bar to assist in preventing over pressure. The safety valve closes after relieving the over pressure. Safety valves can be become clogged and therefore do not guarantee complete protection.

The user must ensure there are adequate safety systems to prevent reactor temperature exceeding maximum allowable working temperature as displayed on the reactor. The maximum temperature applies to the jacket fluid temperature and the contents.

Fast temperature changes (thermal shock) must be avoided to prevent glass fractures. The maximum permissable value for fast, shock like temperature changes is 30°C. Do NOT fill hot glassware with cold fluid, or cold glassware with hot fluid.

The maximum permissable temperature difference between jacket fluid temperature and contents is 30°C. The maximum permissable rate of heating and cooling of the reactor jacket is 5°C/min.

Under normal usage this device reaches high temperatures and may therefore cause burns. Exercise caution when touching the vessels and other attached components.

Always connect the instrument to an earthed AC power outlet. The operating voltage is indicated on the specification sticker. Non observance of this provision may result in damage to the Atlas module or in personal injury or damage to property.

2.1.4 Pressure and Temperature Limits

- Maximum Allowable Working Pressure: +3 barg
- Minimum Pressure: -1 barg
- Maximum Temperature (Jacket): +200°C
- Minimum Temperature (Jacket): -80°C

2.1.5 ATEX

This product does NOT comply with the ATEX directive and must NOT be used in explosive atmospheres.

2.1.6 Cleaning

Cleaning should only be performed by personnel trained in such work, and who are aware of the possible dangers involved. Atlas (and all the associated hardware) has not been designed for sterilization or use with an autoclave.

Atlas may contain hot components, allow the unit to cool before performing any cleaning operations.

The On/Off switch must be in the OFF position and the unit disconnected from both mains and any attached apparatus.

Disconnect gas inputs and ensure any pressure in the system is safely vented.

It is of extreme importance to prevent damage to the pressure systems when cleaning, in particular scratching/chipping glass parts.

Please see Section 10 Cleaning Systems for detailed information about cleaning systems

2.1.7 Maintenance

Maintenance should only be attempted by qualified service personnel or under guidance by Syrris.

Atlas may contain hot components, allow the unit to cool before performing any maintenance operations.

The On/Off switch must be in the OFF position and the unit disconnected from the mains and any apparatus.

Disconnect gas inputs and ensure any pressure in the system is safely vented before performing any maintenance operations.

The safety manifold including pressure relief valve operation must be checked as part of a scheduled maintenance program. See Section 9.1.1 Atlas Potassium 3 Bar Pressure Relief Valve Test Protocol.

Under normal operating conditions, the rupture disc should be replaced annually. Severe operating conditions may require that the rupture disc be replaced more frequently.

In the event of a vessel rupture, the system needs to be decontaminated and returned to Syrris for evaluation

2.1.8 Static discharge

To avoid the build-up of static within the unit, which could provide a source of ignition, Atlas is fitted with an earth bonding point (indicated on the rear of the unit). This earth bonding should be checked to ensure that static build-up is not possible as part of a risk assessment.

2.2 Returning Equipment

Equipment which has been contaminated with, or exposed to, body fluids, toxic chemicals or any other substance hazardous to health must be decontaminated before it is returned to Syrris or its distributor. Complete the Syrris decontamination certificate (See section 12.2) and return with the equipment.

2.3 Environmental Conditions

Usage For indoor use only

Temperature range: 5°C to 40°C

Humidity: Maximum relative humidity of 80%

2.4 Waste Electrical and Electronic Equipment (WEEE) statement

Syrris is compliant with the EU directive 2002/96/EC on waste electrical and electronic equipment (WEEE) please contact info@syrris.com for directions and information on end-of-life policy.

Notes, Notices, and Cautions

NOTE: A NOTE indicates important information that helps you make better use of your atlas.

NOTICE: A NOTICE indicates either potential damage to hardware or loss of data and tells you how to avoid the problem.

CAUTION: A CAUTION indicates a potential for property damage, personal injury, or death.

3 Hardware Overview

This section is designed to give an overview of the hardware used in the Syrris 3 Bar pressure systems. The hardware overview will include the configuration of parts and explanation on individual components specifically required for the Atlas Potassium 3 Bar Pressure System and Globe 3 Bar Pressure System.

The following Reactor Systems are covered in this manual:

- Atlas Potassium 3 Bar Pressure System
    - Automated glass jacketed reactor system with a volume range of 100ml to 3L, designed for pressure applications up to 3 bar.
- Globe 3 Bar Pressure System
    - Manual glass jacketed reactor system with a volume range of 100ml to 3L, designed for pressure applications up to 3 bar.

NOTE: The instructions and configuration of the Globe 3 Bar Pressure System are identical to the Atlas Potassium 3 Bar Pressure System. This manual will therefore refer mostly to the Atlas Potassium 3 Bar Pressure System with instructions.

Atlas is a very flexible platform and multiple systems can be configured.

Please see separate manuals for:

Atlas Systems: Automate processes from -80°C to +250°C and from vacuum to 0.25bar. Automate temperature control and overhead stirring from 50ml to 5L.

(Batch Reactor Manual 1 – Atlas Systems (2000255)).

Software controlled systems: Add PC Software to enable complex recipe control, real time data plotting and control of third party hardware.
(Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (2000262))

Atlas Sodium High Pressure System: Stainless steel (or Hastelloy) reactor system with a volume range of 100ml to 450ml, designed for pressure applications up to 200 bar (100 bar with Hydrogen).
(Batch Reactor Manual 10 – Atlas Sodium High Pressure Systems (2000287))

Atlas Volumetric Dosing: Add the Atlas Syringe Pump for versatile volumetric dosing that consists of two syringes e.g. dose many reactors at a time or have temperature dependant dosing.
(Batch Reactor Manual 3 – Atlas Syringe Pumps (2000257)).

Atlas Gravimetric Dosing: Pump a defined mass of reagent from a balance to a reactor via a controlled peristaltic pump.
(Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (2000262))

Atlas pH Control: Control the pH of a reaction by adding either acid and/or base as necessary to maintain a set pH value.
(Batch Reactor Manual 3 – Atlas Syringe Pumps (2000257)).

Atlas Sonolab: Use ultrasound energy in crystallizations for narrow particle size distribution and polymorph control.
(Batch Reactor Manual 4 – Atlas Sonolab (2000258))

Atlas FTIR Analysis: Monitor reactions in real time with an integrated mid-IR spectrometer.
(Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (2000262))

Atlas Calorimeter: Plot power and enthalpy in real time using heat flow or power compensation calorimetry.
(Batch Reactor Manual 2 – Atlas Calorimeter (2000256))

Atlas Parallel: Automate up to 8 systems in parallel from one PC using the Atlas Parallel software.
(Batch Reactor Software Manual 3 – Parallel Software (2000264))

For more information about any of these systems please email:
info@syrris.com

3.1 Atlas Potassium 3 Bar Pressure System Hardware Overview

The Atlas Potassium 3 Bar Pressure System is a modular 3 bar pressure reactor system that can be controlled automatically or manually. It is available with reactor volumes of 100ml, 250ml, 500ml, 1L, 2L or 3L. All reactors can be changed in under a minute without the need for tools.

The Atlas Potassium 3 Bar Pressure System is designed for applications where elevated pressures are required (e.g. hydrogenations, carbonylations etc.) or high vacuum applications. The basic system allows automatic temperature control (-80°C to +200°C)*, stirrer control up to 800rpm (or 2000rpm with appropriate upgrade) and pressure sensing. All data is displayed and automatically logged by the base unit. The system can be controlled either manually or in an automated manner using Atlas Software. The system includes a 3 Bar System Safety Manifold containing a Rupture (burst) Disc and Pressure Relief Valve. A safety enclosure is an important standard module in 3 bar pressure systems. This safety enclosure has been designed to assist in protecting the user in the event of a vessel rupture. The enclosure has been designed to contain large glass fragments and to vent at the rear of the system.

Additional modules can be added to the system configuration to allow additional pressure and flow control options. These additional modules include:

- 3 Bar Gas Pressure Control Module (PN: 2200497)
- Gas Selection Module (PN: 2200375)
- Mass Flow Meters/Mass Flow Controller (see Batch Chemistry Reactors Catalogue for part number details and options)
- 3 Bar Dip Tube for Autosampling (PN: 2200373)
- Automated control using Syrris PC software (see **Batch Chemistry Reactors** Catalogue for part number details and options)

There are multiple ways that the reactor systems can be configured for use, depending on the application. The most common hardware configurations of the Syrris pressure systems include the following modes of operation:

- Manual pressure control
- Automatically maintain a pressure within the reactor
- Pressure control with mass flow monitoring
- Maintain a pressure and monitor mass flow (ideal for gas uptake monitoring)
- Mass flow control
- Off gas monitoring

The Atlas Potassium 3 Bar Pressure System hardware overview is shown in Figure 1 on page 17. The Atlas system allows multiple configurations and upgrade routes. This overview is designed to highlight the components that

are important specifically for the Atlas Pressure System. For further information on complete Atlas systems and options, please see Batch Reactor Manual 1 – Atlas Systems (PN: 2000255).

The components that are specifically incorporated in the Atlas Potassium 3 Bar system that are NOT included in the standard Atlas system (for non-pressure applications) are:

- Jacketed Reaction Vessel (rated to 3 Bar)
- Jacketed Vessel Clamp (3 Bar system)
- Stirrer Seal and Guide Stainless Steel (3 Bar system) / Stirrer Seal and Guide PTFE (3 Bar system)
- Jacketed Vessel Lid (3 Bar system)
- Safety Pressure Manifold
- Atlas 3 bar System Safety Enclosure

NOTE: Standard Atlas vessels are not compatible with the Atlas Potassium 3 Bar system. The Jacketed Vessel Clamp (3 Bar system) will accept only pressure reactors. It is important to understand the difference between the Jacketed Vessel Clamp (3 Bar system) and the standard Jacketed Reactor Clamp to avoid confusion of vessel compatibilities. This is particularly important when there are multiple systems on one site.

The Safety Pressure Manifold is connected to the reactor via the lid and is supported just above the reactor. This manifold is shown separately in Figure 2. It consists of:

- Rupture (burst) disc and assembly.
- Pressure relief valve.
- Manual inlet and outlet valves.
- Analogue pressure gauge

The system can log and display pressure using an optional digital pressure sensor. A picture of the pressure sensor is shown in Figure 3. The pressure sensor is connected to a Pressure Node (see Batch Reactor Manual 1 – Atlas Systems (PN: 2000255) for more information on Atlas nodes and sensors), which is plugged into the Atlas Base unit.

Instructions for how to operate the Gas Pressure Control module and Gas Selection module are not covered in this manual. Please see separate manuals of these modules: Batch Reactor Manual 8 – Gas Pressure Control Module (PN: 2000285) and Batch Reactor Manual 9 – Gas Selection Module (PN: 2000286).

Information on how to construct automated experiments within Atlas and Reactor Master software can be found in the Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (PN: 2000262).

The Globe 3 Bar System assembly and hardware configuration is similar to that of Atlas. All of the pressure parts are identical, which makes the instruction and use of the Globe 3 Bar System very similar to the Atlas Potassium 3 Bar System. Before using the Globe 3 Bar System, please read the Batch Reactor Manual 6 – Globe Systems (PN: 2000260) before reading this manual. The hardware configuration of the Globe 3 Bar System is shown in Figure 4.

*****Refers to Vessel Jacket temperatures. Choices such as circulator fluid, circulator and a low temperature upgrade kit will influence the achievable temperature range.

3.1.1 Atlas Potassium Pressure Reactor Systems hardware

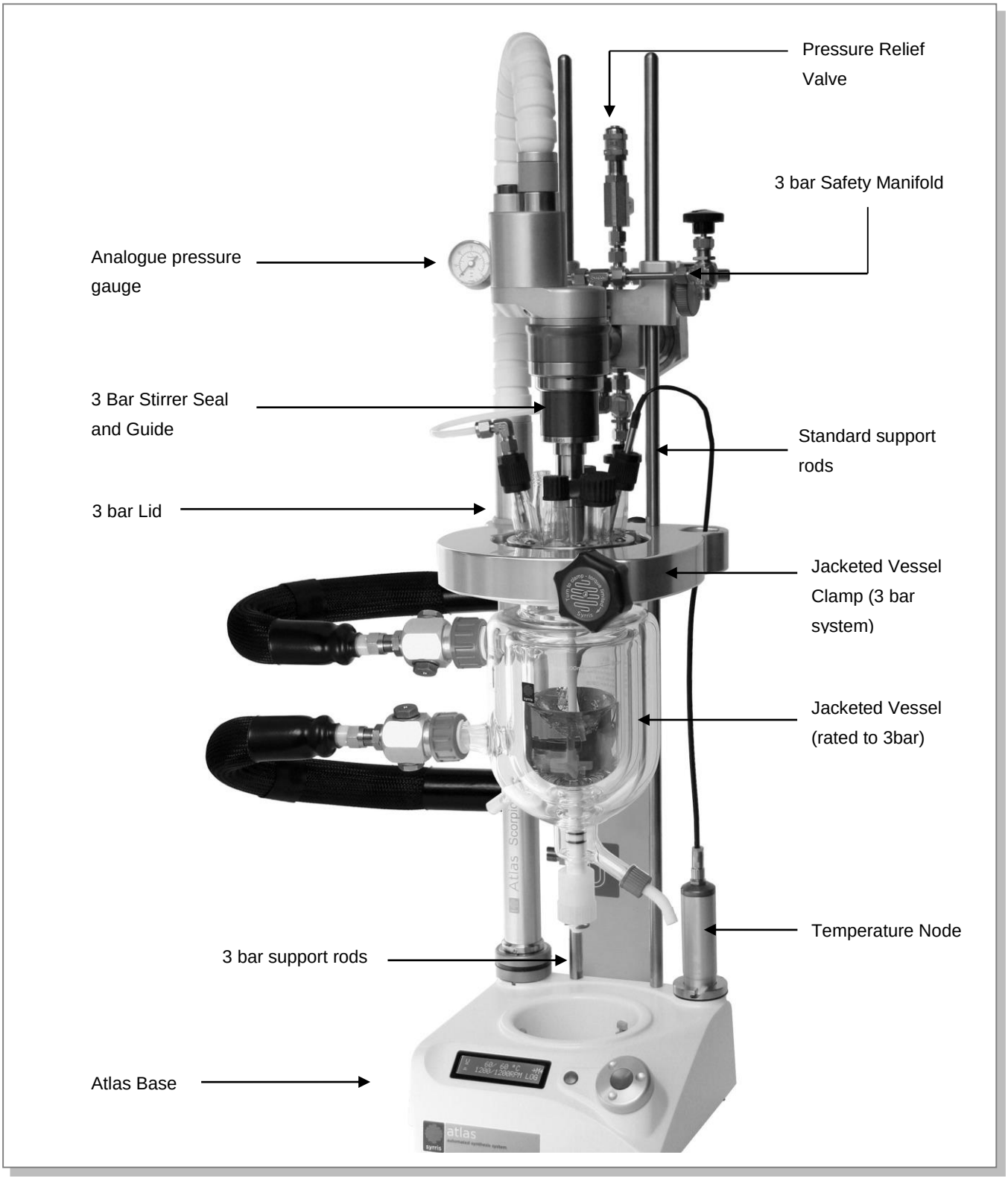


Figure 1

3.1.2 3 Bar Safety Manifold hardware

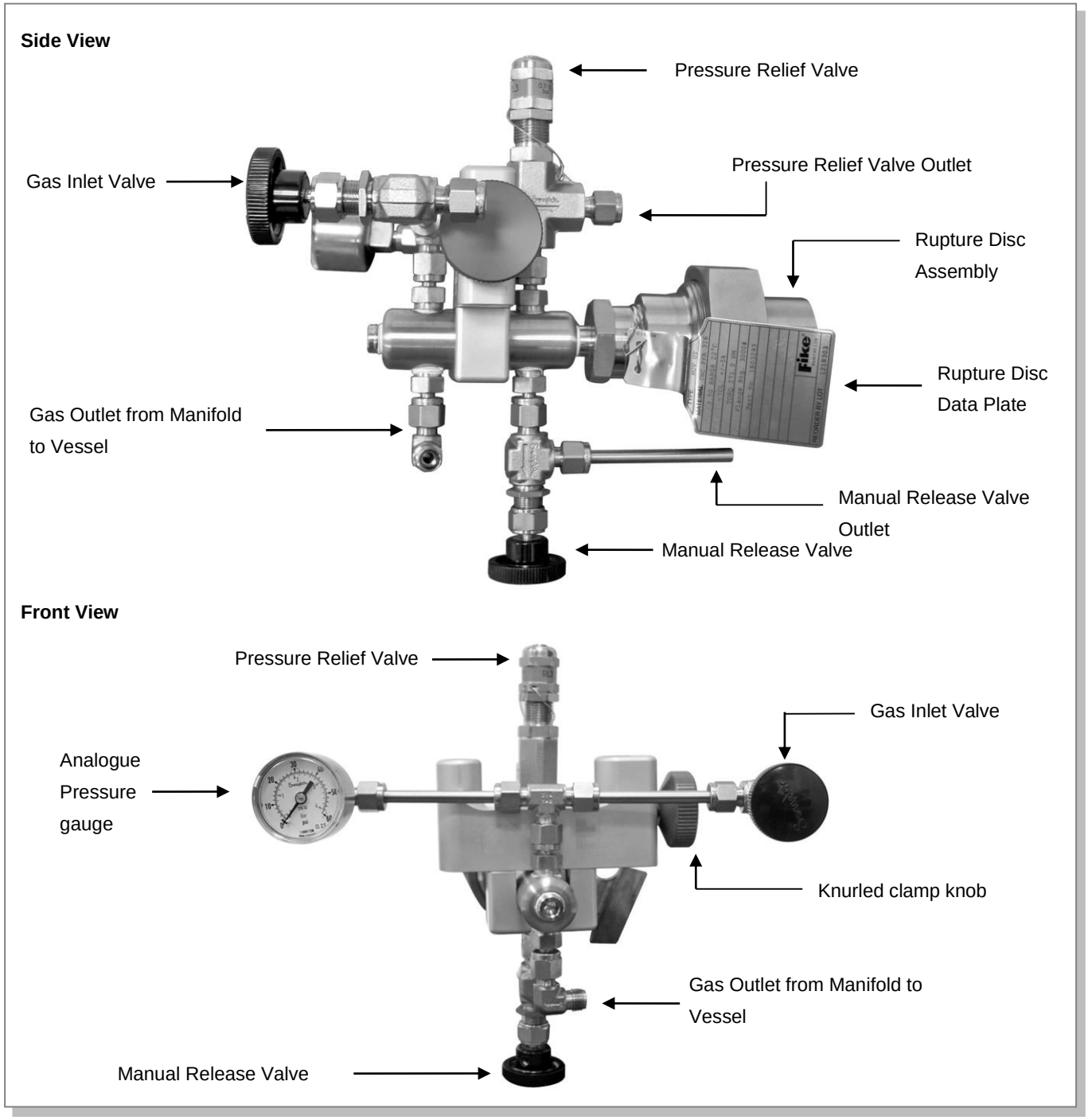


Figure 2

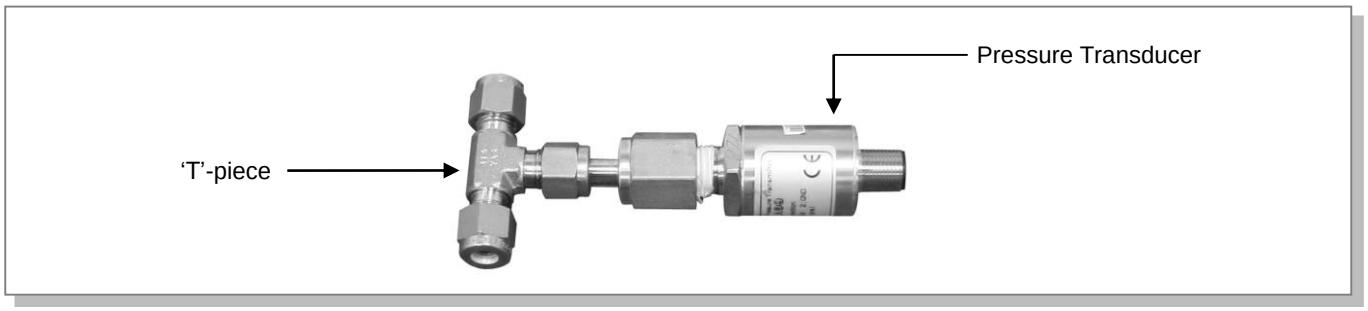


Figure 3

3.1.3 Globe Pressure Reactor Systems Hardware

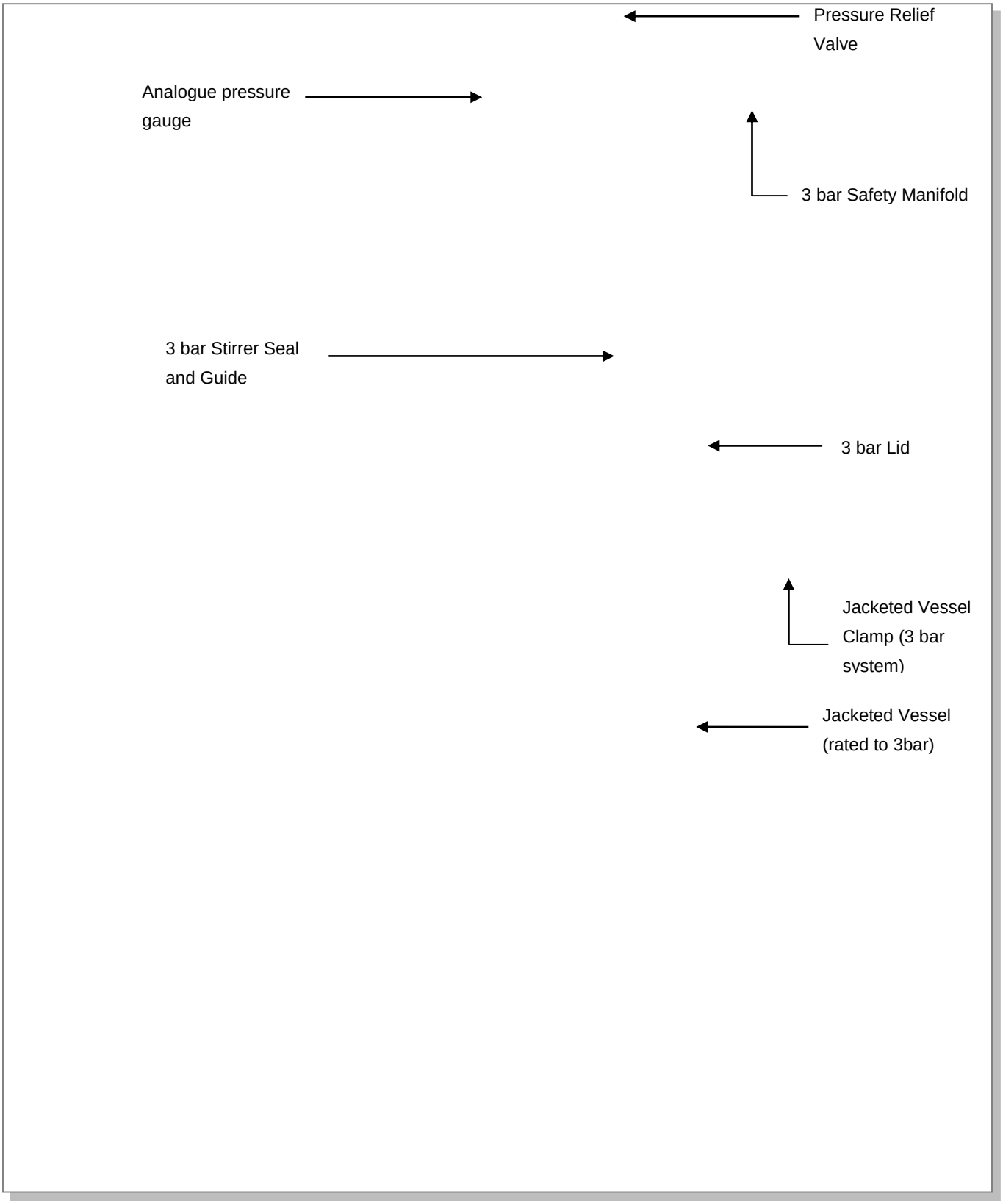

Pressure Relief Valve
Analogue pressure gauge
3 bar Safety Manifold
3 bar Stirrer Seal and Guide
3 bar Lid
Jacketed Vessel Clamp (3 bar system)
Jacketed Vessel (rated to 3bar)

Figure 4

3.1.4 Atlas Potassium 3 Bar Pressure System Specifications

The specifications for the Atlas Potassium 3 Bar Pressure systems are:

Atlas Potassium 3 Bar Pressure System	
Temperature Range	-80°C to +200°C Jacket Temperature All temperature ratings refer to maximum jacket temperatures (not reactor content temperatures).
Overhead Stirring	Up to 800 RPM using Atlas Scorpion and up to 2000 RPM using Heidolph Stirrer Motor
Vessel / Lid Materials of Construction	- Stainless Steel Lid (TS316SS) - PTFE Lid - Borosilicate glass vessel
Vessel Seals	- FFKM
Maximum Working Pressure	- 3 Bar
Maximum vessel jacket pressure	- 1 Bar
Volumes	- 100ml (PN: 2200411) - 250ml (PN: 2200412) - 500ml (PN: 2200332) - 1000ml (PN: 2200331) - 2000ml (PN: 2200413) - 3000ml (PN: 2200414)
Mode of Heating	- External Circulator

3.1.4.1 3 Bar System Safety Manifold

The 3 Bar System Safety Manifold contains:

1. Pressure Relief Valve
2. Rupture (Burst) Disc and Assembly
3. Manual Valves
4. Dial Gauge

The specification of each of these items are listed in this section.

3.1.4.1.1 Pressure Relief Valve

The Pressure Relief Valve is a standard item supplied by Swagelok™, and has the following specification:

Inlet and Outlet Manual Valves	
Swagelok™ Part Number	SS-RL3S4
Swagelok™ Part Description	SS Low-Pressure Proportional Relief Valve
Service Class	Low Pressure
Material of Construction	316 Stainless Steel
End Connection Size	¼" (both connections)
End Connection Type	Swagelok™ tube fitting (both connections)
Manual Override	No
Lubricant Used	Dow Corning Molykote 55 Grease
Seal Material	Fluorocarbon FKM
Maximum Temperature Pressure Rating	275°F at 225psig/135°C at 15.5bar
Room Temperature Pressure Rating	100°F at 225psi /37°C at 15.5bar

This Pressure Relief Valve is set during manufacture to a relief pressure of 2.75bar (-0.05/+0.05). Once this is set and checked, the locknut is connected and wire-locked.

NOTE: Ensure that the Pressure Relief Valve has not been tampered with. This can be seen with inspection of the wire-lock. If the wire-lock has shown signs of tampering, do not use the system. The Pressure Relief Valve must be shipped back to Syrris.

NOTE: The Pressure Relief Valve should only be adjusted by suitably qualified personnel approved by Syrris or its representatives.

3.1.4.1.2 Rupture (Burst) Disc

The Rupture (Burst) Disc is mounted in a 316 Stainless Steel holder fitted to the end of the Pressure Manifold. The holder is a reusable item, but the discs are a onetime fitment only. The discs cannot be reused if:

- They have been removed from the holder
- They have been ruptured

The Burst Disc is supplied by Fike UK Ltd, and has the following specification:

Rupture (Burst) Disc	
Material of Construction	Inconel 600 (top section) PTFE liner 316 Stainless Steel Support
Diameter	1” (rupture disc diameter)
Rupture pressure	3.3bar (+/- 5% tolerance) at 22°C
Rupture pressure range	3.135 to 3.465bar at 22°C

NOTE: The rupture disc temperature is dependent on many factors including (but not limited to) ambient temperature, jacket temperature, reactor temperature, fill volumes and reactor contents.

3.1.4.1.3 Valves

The Inlet and Outlet Manual Valves are supplied by Swagelok™, with the following specification:

Inlet and Outlet Manual Valves	
Swagelok™ Part Number	SS-1R34-A
Swagelok™ Part Description	SS Integral Bonnet Angle-Pattern Needle Valve, ¼” Swagelok™ Tube Fitting, Regulation stem
Flow Pattern	Angle Pattern (2-way)
Material of Construction	316 Stainless Steel
End Connection Size	¼” (both connections)
End Connection Type	Swagelok™ tube fitting (both connections)
Handle	Phenolic Knob (Black)
Lubricant Used	WL7 consisting of Perfluorinated Polyether, PTFE, Tungsten Disulfide
Maximum Temperature Pressure Rating	450°F at 3435psig/232°C at 236bar
Room Temperature Pressure Rating	100°F at 5000psi /37°C at 344bar

There are two valves on the 3 Bar System Safety Manifold (Figure 2). The valve located in the upper position is the Inlet Valve. The valve located in the lower position is the Outlet (Manual Release) Valve.

3.1.4.2 Stirrer Seal and Guide (3 Bar System)

The Stirrer Seal and Guide connects to the Lid and contains a magnetically coupled drive to allow for overhead stirring under pressure/vacuum.

	Stirrer Seal and Guide Stainless Steel (3 Bar System) (PN: 2200334)	**Stirrer Seal and Guide PTFE (3 Bar System) (PN: 2200405)**
Material of Construction	316 Stainless Steel	316 Stainless Steel PTFE
Wetted Materials (inside reactor)	316 Stainless Steel	PTFE
Seals Material	FFKM	FFKM

There are two O-ring seals in both Stirrer Seal and Guide parts. One seal is situated at the connection point to the 3 Bar Lid. The other seal is situated internally in the upper magnetic drive. These seals are static seals and should not normally suffer from wear and tear.

When removing the magnetic drive from the lid, take care not to damage the O-ring on re-assembly.

The O-rings are installed as radial seals. The magnetic drive will only require hand-tightening into the adapter. The adapter should also only require to be hand-tightened into the lid.

There is no testing to perform on the magnetic drive or adapter in isolation, though Snoop® Liquid Leak Detector may be used to check for leaks when the unit is mounted on an operational system.

4 Quick Start Guides

Quick start guide instructions are included in this manual and are only intended for users familiar with using the Atlas Sodium High Pressure System. Prior to use on any Syrris Pressure System, the user must comply with all safety and accident prevention regulations and operate in full compliance with local safety codes and rules.

4.1 Atlas Potassium 3 Bar Pressure Quick Start Guide

1. Assemble the Atlas Potassium 3 Bar Pressure System (Figure 5) by following the instructions in section 5.1 of this manual.
2. Add sensors (Probes and Nodes) by following the instructions in section 6.2 of the Batch Reactor Manual 1 – Atlas Systems (PN: 2000255)
3. Set up and connect the Circulator by following the instructions in section 6.9 of Batch Reactor Manual 1 – Atlas Systems (PN: 2000255)
4. Switch on the Base Unit using the ON/OFF switch.
5. Read section 4.1 of Batch Reactor Manual 1 – Atlas Systems (PN: 2000255) to understand the start-up routine, the home screen and how to operate the control knob.
6. Follow instructions in section 7.3 of the Batch Reactor Manual 1 – Atlas Systems (PN: 2000255) to use manual control from the Base Unit. This is the quickest way to control the Atlas Potassium 3 Bar Pressure System.
7. For more detailed information or advanced functionality read this manual in detail.

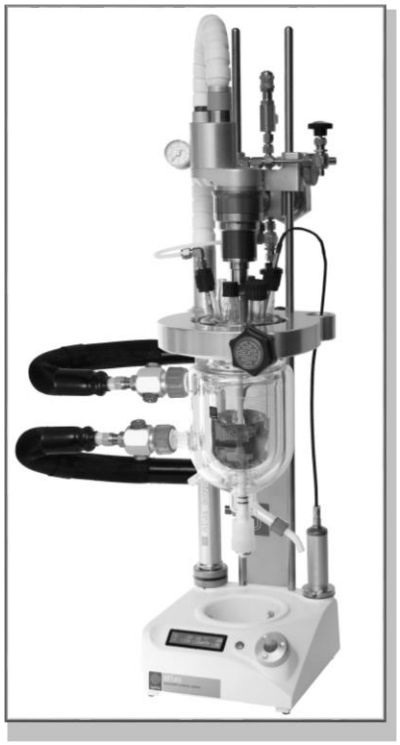


Figure 5

5 Connecting, Setting Up Systems and Physical Use

This section explains how to set up both the Atlas Potassium 3 Bar Pressure System and the Atlas Sodium High Pressure System. One of the key features of the Atlas is the modularity of the system and it is very important to understand how the modules connect together.

5.1 Assembling the Atlas Potassium 3 Bar Pressure Systems

This section explains how to assemble the Atlas Potassium 3 Bar Pressure Systems. This section contains information that will be useful when you are connecting and setting up the system. The system configuration is similar to the Atlas Potassium standard system. Please ensure you are familiar with the Atlas Potassium configuration in Batch Chemistry Manual 1 – Atlas Systems (PN: 2000255).

5.1.1 Connect the Support Rods, Oil Drain Unit and Scorpion Overhead Stirrer Pole to the Base Unit

Connect the Support Rods, Oil Drain Unit, Oil pipes to the Oil Drain Unit and Overhead Stirrer Pole to the Base Unit as per section 6.2 of Batch Reactor Manual 1 – Atlas Systems (PN: 200025).

(NOTE: The Oil Drain Unit and Safety Enclosure have purposely been omitted from the following assembly photographs for clarity)

Figure 6

5.1.2 Assemble the 3 bar Safety Enclosure onto the Base Unit

NOTE: Alternative Support Rods are supplied with the 3 bar Safety Enclosure, which have a different thread to the standard Support Rods. Before assembling the 3 bar enclosure onto the Base Unit, ensure the correct Support Rods have been installed. These poles have a threaded aperture on the top surface. Figure 6

NOTE: Ideally two users are required for this assembly.

1. Feed the flexible white section of the Scorpion Overhead Stirrer pole into the plastic guides on the Safety enclosure. Feed it though the guide on the bottom of the enclosure first.
2. Continue to feed the white section of the Scorpion Overhead Stirrer pole through the second guide at the top of the enclosure (NOTE: Do not slide the enclosure onto the Support Rods at this point (see figure 7.1)).
3. Position the Oil Drain Unit over the Support Rods in conjunction with the 3 bar Safety Enclosure and slide the two items down together as shown in Figure 7.2
4. Ensure the Oil Drain Unit and 3 bar Safety Enclosure are fully seated on the Support Rods with a small protrusion of the Support Rods being visible out of the top of the Oil Drain Unit as shown in Figure 7.3.

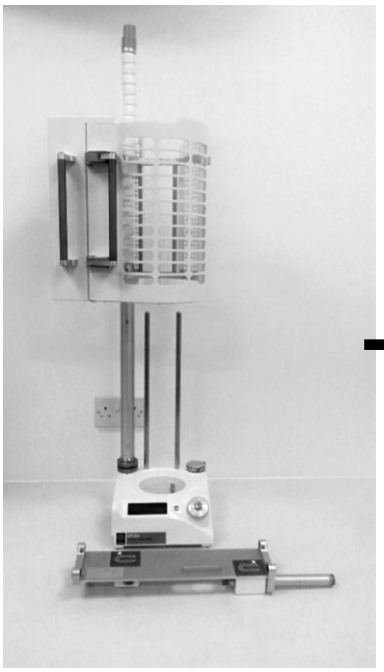


Figure 7.1

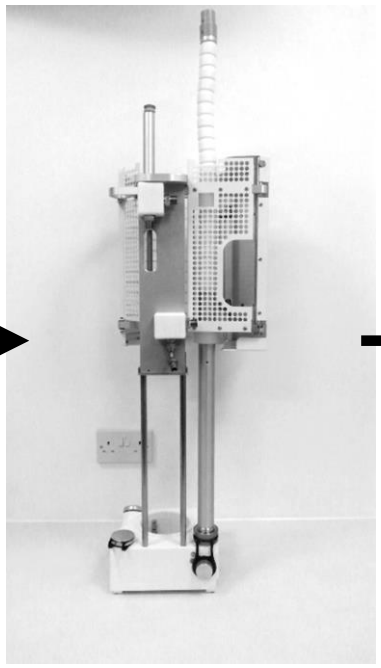


Figure 7.2

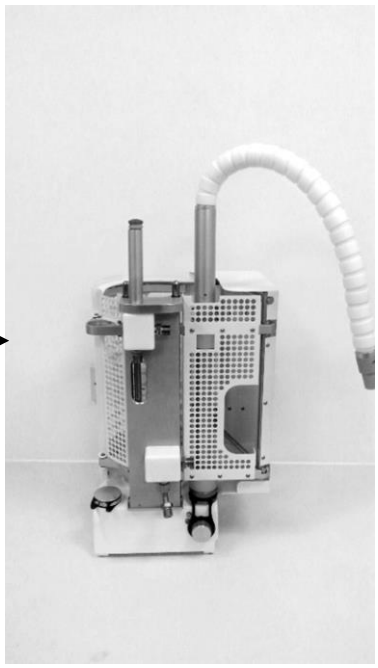


Figure 7.3

Figure 7

5.1.3 Connect the Jacketed Vessel Clamp (3 Bar System)

The Jacketed Vessel Clamp (3 Bar System) is a high-force clamping mechanism for Atlas Potassium 3 Bar Pressure and Globe 3 Bar systems.

1. Feed the Jacketed Vessel Clamp (3 Bar System) over the end of the Scorpion Overhead Stirrer pole (Figure 8).
2. The beige end of the Scorpion Overhead Stirrer pole goes into the beige hole in the corner of the Jacketed Vessel Clamp (3 Bar System) (from the underside).
3. Slide the Jacketed Vessel Clamp (3 Bar System) down the white flexible section of the Scorpion Overhead Stirrer pole until it seats in position on top of the Support Rods and Scorpion Overhead Stirrer (Figure 9).
4. The metal dowel pin on the metal upright of the Scorpion Overhead Stirrer Pole must locate in the slot located in the beige (PEEK) plastic part on the underside of the Jacketed Vessel Clamp (3 Bar System).

Figure 8

Figure 9

5.1.4 Connect the Secondary Support Rods to the Base Unit

The Pressure Vessel Clamp has connections for the Secondary Support Rods on the upper surface.

The clamping bolts for the 3 bar Safety Enclosure must be screwed though the Jacketed Vessel Clamp onto the lower Support Rods (Figure 10).

1. Screw the Secondary Support Rods into their connection tightly until they stand vertically and cannot be pushed left or right (Figure 11).
2. Use a spanner on the flattened section at the base of the Secondary Support Rods to tighten fully.

Figure 10

Figure 11

5.1.5 Connect the 3 Bar System Safety Manifold to the Secondary Support Rods

The 3 bar Safety Manifold attaches to the Secondary Support Rods via a clamp mechanism.

1. With the red knurled knob fully unwound, slide the Clamp over the Secondary Support Rods. The two Secondary Support Rods locate through the two holes in the Clamp (Figure 12).
2. Lower the Clamp and Manifold assembly down the Secondary Support Rods until the Manifold is situated approximately half-way down the Secondary Support Rods (Figure 13).

3. Whilst supporting the weight of the 3 bar Safety Manifold with one hand, screw the red knurled knob on the right of the clamp to lock the 3 bar Safety Manifold in place.

5.1.6 Attach the Stirrer Seal and Guide Stainless Steel (3 Bar System) to the Potassium Pressure Vessel Lid

The Stainless Steel Stirrer Seal and Guide Stainless Steel (3 Bar System) (Figure 14) screws into the Jacketed Vessel Lid (3 Bar System). The seal is made on the stirrer seal and guide via a radial seal from the FFKM 'O'-ring.

1. Place the Stainless Steel Stirrer Seal and guide through the threaded aperture in the Potassium Pressure Vessel Lid.
2. Rotate the Stainless Steel Stirrer Seal and Guide clockwise whilst holding the Potassium Pressure Vessel lid stationary. (Figure 15)
3. The black 'O'-ring should be hidden between the Stainless Steel Stirrer Seal and Guide and the Potassium Pressure Vessel lid when it is correctly installed (NOTE: Take care not to damage the 'O'-ring when screwing the Stirrer Seal and Guide into the Potassium Pressure Vessel lid).

Figure 12

Figure 13

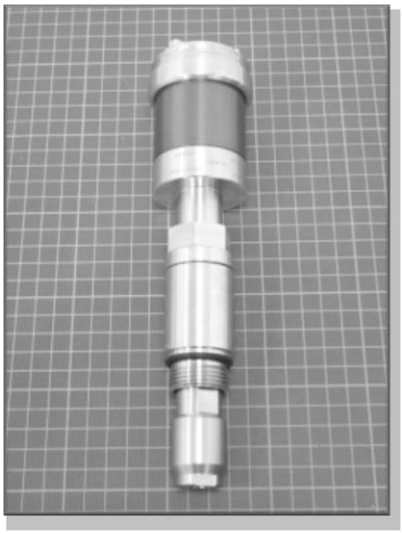


Figure 14

Figure 15

5.1.7 Attach the Stirrer into the Stirrer Seal and Guide Stainless Steel (3 Bar System) and Jacketed Vessel Lid SS (3 Bar System)

The stirrer rod shaft fits into the Stainless Steel Stirrer Seal and Guide and is held in place with a gripping chuck attachment.

1. Rotate the gripping chuck attachment anti-clockwise to unscrew until three or four thread turns are visible. (do not unscrew completely) (Figure 16).
2. Push the stirrer fully into the unscrewed chuck attachment. The stirrer shaft will be supplied pre-cut to the correct length for the supplied pressure reactor.
3. Rotate the gripping chuck attachment clockwise until the stirrer shaft is fully gripped (Figure 17).
4. Use spanner to tighten and release if necessary.

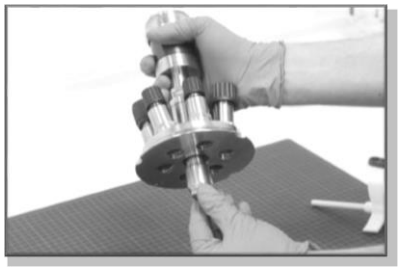


Figure 16

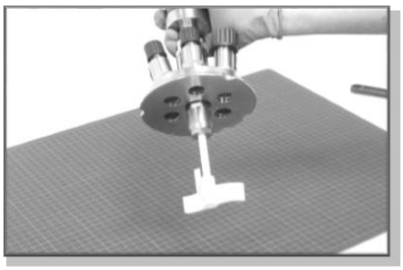


Figure 17

NOTICE: When the System is being prepared for shipment the stirrer shafts are optimally cut for each vessel. All vessel sizes must have a corresponding stirrer.

5.1.8 Position the Stirrer, Stirrer Seal and Guide Stainless Steel (3 Bar System) and Jacketed Vessel Lid SS (3 Bar System)

1. Place the Jacketed Vessel Lid SS (3 Bar System), Stirrer Seal and Guide Stainless Steel (3 Bar System) and Stirrer assembly into the Jacketed Vessel Clamp (3 Bar System) (Figure 18).
2. Ensure that the locating lug on the Jacketed Vessel Lid SS (3 Bar) is located into the locating cut-outs on the Jacketed Vessel Clamp (3 Bar System). Ensure that the Jacketed Vessel Lid (3 Bar) is in the desired orientation.
3. When the Jacketed Vessel Lid (3 Bar System) is correctly inserted and aligned, it will sit level.

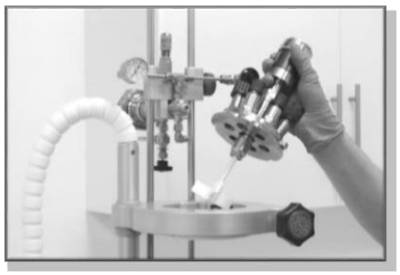


Figure 18

5.1.9 Insert the Jacketed Vessel (Rated to 3 Bar) into the Jacketed Vessel Clamp (3 Bar System)

1. Ensure the Jacketed Vessel Clamp (3 Bar System) is in the unlocked position with the Vessel seal placed in the indent on the top of the vessel.
2. Hold the Jacketed Vessel with the arrow marked “A insert” facing directly to the front (Figure 19).
3. Gently insert the Jacketed Vessel into the Jacketed Vessel Clamp (3 Bar System) from underneath.
4. With the vessel fully inserted until it ‘stops’ and ensuring it is level, rotate the vessel clockwise through 60 degrees (if looking from above).
    a. The two red connections to the oil jacket should then extend to the left and be parallel to the front face of the system.
    b. The Jacketed Vessel now rests on the Pressure Vessel clamp and any support can be removed.
5. The Jacketed Pressure Vessel is now in place but not secured. (Figure 20)

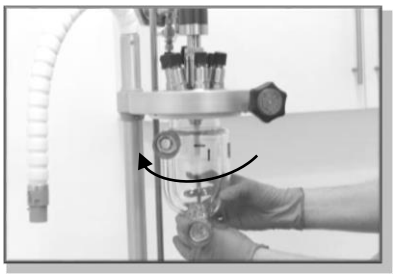


Figure 19

Figure 20

5.1.10 Clamp the Jacketed Vessel (Rated to 3 Bar), Jacketed Vessel Lid SS (3 Bar System) and Stirrer Assembly into position using the Jacketed Vessel Clamp

When correctly inserted in the Jacketed Vessel Clamp (3 Bar System), the Jacketed Vessel and Jacketed Vessel Lid SS (3 Bar System) can be moved or removed independently.

1. Secure the Jacketed Vessel and Jacketed Vessel Lid by rotating the knob on the Jacketed Vessel Clamp in a clockwise direction. Arrows are present on the locking knob to confirm tightening direction (Figure 21).
2. Continue rotating the locking knob until it ‘slips’.
3. If rotation is continued the knob will ‘slip’ further as the correct torque has been achieved. This is signified by a clicking of the knob.
4. Once the knob ‘slips’ further the vessel is ‘locked’ in position.

Figure 21

5.1.11 Install probes into the Jacketed Vessel Lid SS (3 Bar System)

All probes on a 3 Bar System come with black pressure rated probe holders with PTFE inserts as shown in Figure 24.

1. Insert the probe into the appropriately sized black pressure rated probe holder (ensuring the orientation of the associated PTFE inserts).
2. Insert the probe and probe holder assembly into the port on the Jacketed Vessel Lid.
3. Tighten the probe holder (turn clockwise) hand tight to achieve a good seal and to prevent any slippage of the probe upon system pressurisation.

Please note the following instructions for glass probes:

PH probe – From the point where the black pressure rated probe holder starts to give some resistance rotate the cap a further half a turn to seal. This equates to a torque value of 3.4N/m.

Turbidity probe – From the point where the pressure rated probe holder starts to give some resistance rotate the cap a further quarter turn to seal. This equates to a torque value of 2.3N/m.

Caution: Never use tools for tightening probe holders. This can cause damage to the probes, pressure rated probe holders and the lid.

5.1.12 Attach the Gas Outlet from the Safety Manifold to the Pressure Vessel Lid

All of the connections on the Safety Manifold are standard ¼" OD Swagelok™ connectors. To connect the 3 Bar System Safety Manifold to the Jacketed Vessel Lid SS (3 Bar System):

NOTICE: To remove either the Jacketed Vessel (Rated to 3 Bar) or Jacketed Vessel Lid (3 Bar System), simply rotate the locking knob fully in an anti-clockwise direction until some resistance is felt. (the opposite direction of the arrow).

1. Remove a black pressure cap from the Jacketed Vessel Lid (SS) (3 Bar System) (which pressure cap is unimportant).
2. Attach the length of ¼" OD PTFE tubing to the Gas Outlet from the Safety Manifold utilising the provided Swagelok™ ¼" fitting (Figure 22).
3. Attach the other end of this tubing to the Swagelok™ elbow and black gas inlet fitting provided (ensuring the internal ferrules are in the correct orientation).
4. Tighten these fitting finger-tight only.
5. Place the black inlet fitting onto the lid and finger-tighten (Figure 23)
6. Tighten the Swagelok™ fittings with a spanner.

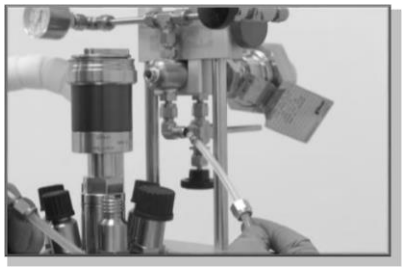


Figure 22

NOTE: The orientation of the internal ferrules of the black gas inlet fitting is shown in Figure 24.

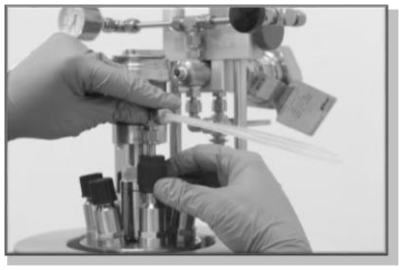


Figure 23

5.1.13 Attach the Gas Outlet from the Jacketed Vessel Lid SS (3 Bar System) to the Gas Pressure Control Module

The 3 Bar Gas Pressure Control Module (2200497) is an optional module which allows the pressure to be actively controlled. If this option is present, connect the Jacketed Vessel Lid to the outlet of the Gas Pressure Control Module.

For further instructions on the set-up and use of the 3 Bar Gas Pressure Control Module please see Batch Chemistry Manual 8 – Gas Pressure Control Module Manual (PN: 2000285).

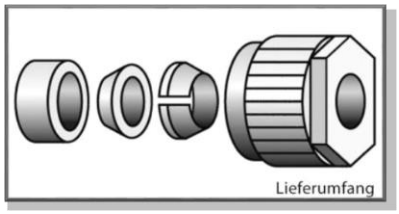


Figure 24

1. Remove one of the black pressure caps from the Pressure Vessel Lid (which pressure cap is unimportant) (Figure 25).
2. Insert the stainless steel outlet attachment assembly (ensuring the internal ferrules of the fitting are in the correct orientation).
3. Tighten the black fitting finger tight only.
4. Slide the tubing in the fitting to the desired depth.
5. Tighten black nut fully (do not over-tighten) (Figure 26).
6. Attach the gas outlet to the outlet position of the Gas Pressure Control Module.
7. Attach the Manual Release Valve Outlet tubing to a suitable exhaust system.

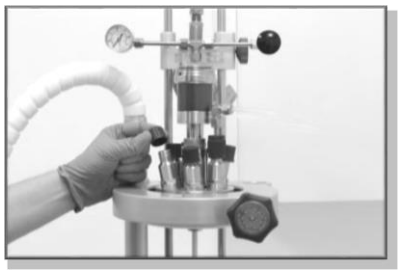


Figure 25

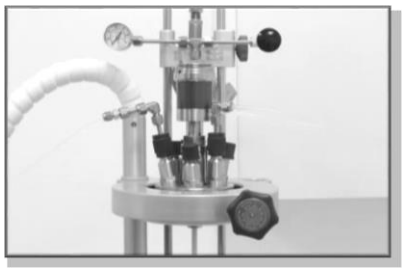


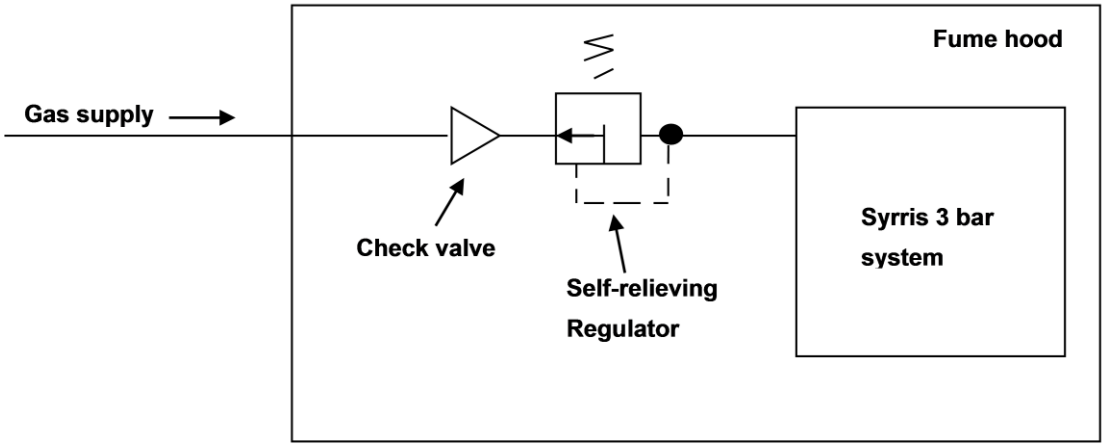
Figure 26

NOTE: If the Pressure Control Module is not utilised, no outlet connection should be made. Venting of the vessel should be carried out via the Manual Release Valve (see 5.2.5 Depressurizing the System).

5.1.14 Attach the Gas Input Source to the Gas Inlet Valve on the 3 Bar System Safety Manifold

Connect the gas input source to the Gas Inlet Valve utilising 1/8” tubing. Ensure that the maximum input pressure is less than the Jacketed Vessel Maximum Allowable Working Pressure (MAWP). For the 3 Bar Systems, the MAWP is 3 Bar.

Caution: If external pressure feed is cut off then the pressure from the reactor could vent backwards towards the supply. If corrosive gasses are present then this could damage your regulator. To prevent this, a check valve and self-relieving regulator can be added to the gas feed as shown in the diagram below.



Caution: If poisonous or corrosive gas is being used to pressurise the reactor it is the user’s responsibility to take precautionary measures to ensure safety. Precautions should be taken to avoid leaks and/or breaks in the gas feed tubing.

5.1.15 Connect the Scorpion Overhead Stirrer Pole to the Scorpion Overhead Motor

1. Align the plug on the end of the Scorpion Overhead Stirrer Pole to the socket on the Scorpion Overhead Stirrer Motor. There is a notch in the Scorpion Overhead Stirrer and a corresponding gap in the socket on the Scorpion Overhead Stirrer Motor to allow the two modules to click together (Figure 27).
2. Push the Scorpion Overhead Stirrer Pole into the Scorpion Overhead Stirrer Motor, then turn ¼ clockwise. The red metal button should pop back into its original position with an audible 'click' when the plug is correctly inserted (Figure 28).
3. The Scorpion Overhead Stirrer has an ON/OFF switch. Press the small red button located next to the stirrer shaft. When the Base unit is on, the Scorpion Overhead Stirrer Pole and Motor are correctly connected, and the on/off switch is pressed, the switch illuminates. Check the button is illuminated when powered on.

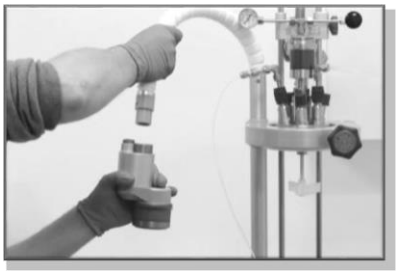


Figure 27

Figure 28

5.1.16 Connect the Overhead Stirrer to the Stirrer Seal and Guide Stainless Steel (3 Bar System)

1. Place the Overhead Stirrer over the Stirrer Seal and Guide Stainless Steel (3 Bar System) (Figure 29).
2. The Scorpion Overhead Stirrer Motor clicks into place over the top of the Stirrer Seal and Guide Stainless Steel (3 Bar System).

Figure 29

NOTICE: To eject the Scorpion Overhead Stirrer Motor from the Stainless Steel Stirrer Seal and Guide, lift the red ring around the Scorpion Overhead Stirrer Motor, this will break the connection and allow the Scorpion Overhead Stirrer Motor to be lifted out of position.

5.1.17 Connect the Oil Pipes from Reactor to Oil Drain Unit

1. Ensure the Pressure Vessel is in the locked position using the Pressure Vessel clamp before connecting the quick connections. See section *5.1.10 Clamp the Jacketed Vessel (Rated to 3 Bar), Jacketed Vessel Lid SS (3 Bar System) and Stirrer Assembly* into position using the Jacketed Vessel Clamp.
2. Ensure that the Oil pipes have a black 'O'-ring seal in place; this prevents damage to the glass Pressure Vessel (Figure 30).
3. Attach the connectors on the Oil Pipes (*Oil Pipes from Reactor to Oil Drain Unit (Pack of 2) PN: 2101014*) to the quick release oil connections on the Jacketed Pressure Vessel (Figure 31).
4. Do not over-tighten the quick connectors onto the side arms of the Pressure Vessel or glassware breakages can occur.

Figure 30

5.1.18 Connect the Oil Pipes from Circulator to Oil Drain Unit to the Circulator

1. Connect the free ends of the Oil Pipes (Oil Pipes from Circulator to Oil Drain Unit (Pack of 2) (PN: 2101015)) from the Oil Drain Unit to the circulator using the M16 connections.

Figure 31

5.1.19 Connect the Optional Digital Pressure Node to the Atlas Base

The Digital Pressure Sensor (2200370) connects directly to a Pressure Node (2200369).

1. Attach the supplied cable to the rear of the Pressure Sensor (Figure 3) to the connector on the Pressure Node.
2. Place the Pressure Node into the Node Socket of the Atlas Base unit.
3. The Atlas Base will recognise the Pressure Node on start-up and display the pressure on the front LCD display, similar to all other Syrris nodes.

Please refer to the Batch Reactor Manual 1 – Atlas Systems (PN:200025) for instructions on Syrris Nodes and the Atlas Base unit.

5.2 Operation of the Atlas Potassium 3 Bar Pressure System

It is very important to follow the pressurization and depressurization protocol exactly. This is to maintain a safe working procedure whilst utilizing the Atlas Potassium 3 Bar Pressure System. Please be familiar with the overall

hardware overview of the system (see Figure 1) and the overview of the 3 Bar System Safety Manifold in Figure 2 and the figure below (Figure 32) which shows where the components are situated to perform the following operations.

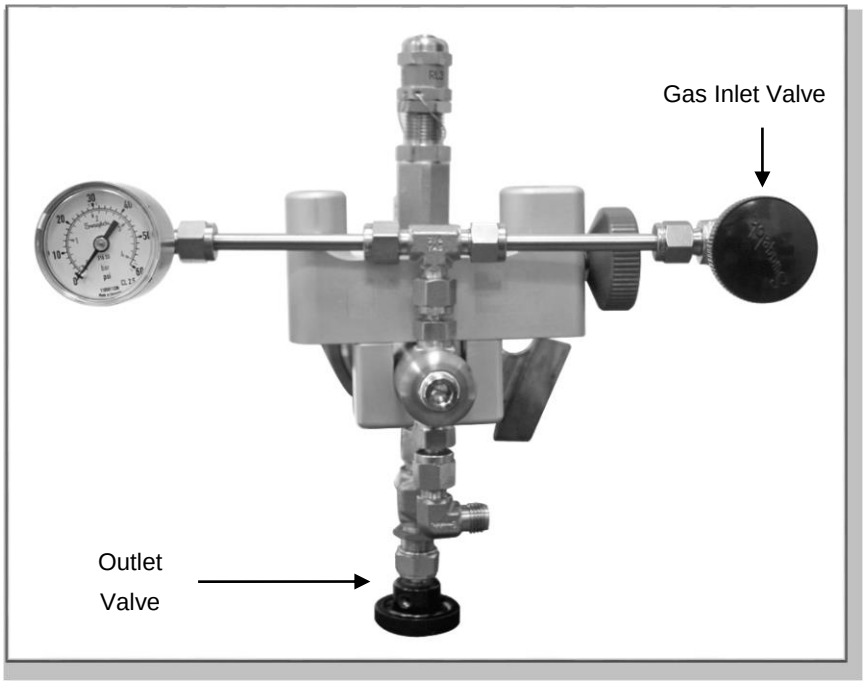


Figure 32

Outlet Valve on the Safety Manifold. The system can be configured in many different ways, depending on the application, mode of operation and modules included in the system. In the simplest form, the system will always consist of:

- An external gas supply
- Gas Inlet Valve (on the Safety Manifold)
- Gas Outlet Valve (on the Safety Manifold

5.2.1 Use of the safety enclosure

The 3 bar Safety Enclosure has been designed to assist in protecting the user in the event of a vessel rupture. The enclosure has been designed to contain large glass fragments and to vent at the rear of the system.

⚠

CAUTION: The safety enclosure alone does not guarantee full protection in the event of vessel rupture.
The system must be installed in a suitable structure. The base, rear, side and top walls must be solid and secure, for example a suitably built and sized fume cupboard.

The front section must be enclosed with a solid and secure screen such as a shatter proof sash door.

Note that all other safety instructions apply. It is the responsibility of the user to assess the suitability of the set up, including safety barricades and screens.

⚠ CAUTION: In the case of spillage of solvent and/or reaction media onto the 3 Bar Safety Enclosure screen the structural integrity may be compromised and therefore, it must be replaced immediately. Please contact Syrris support (support@syrris.com) for details on how to purchase and install a replacement screen.

Operation

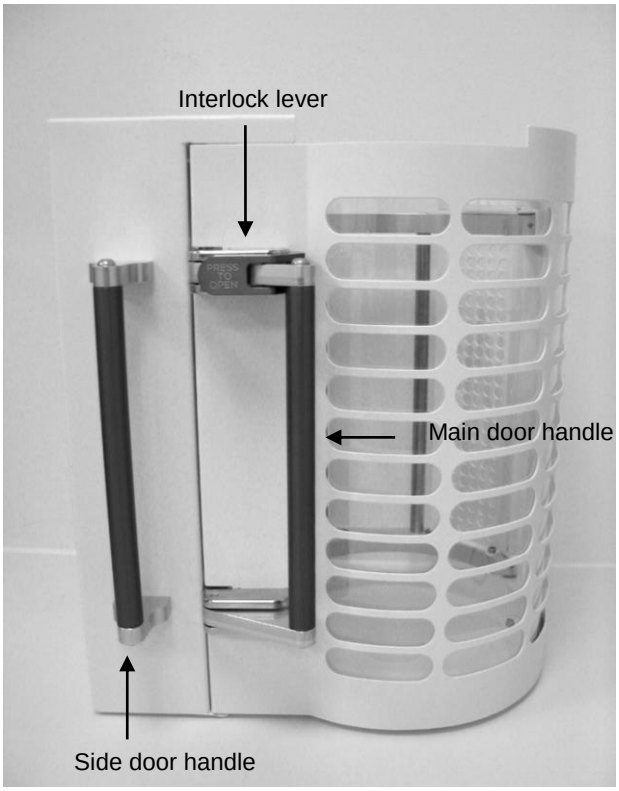


Figure 33

To open the main door of the 3 bar Safety Enclosure, press the interlock lever in with your thumb to unlock the door (Figure 33.1) and then pull the handle of the main door towards you (Figure 33.2), which will open the handle, and then, in turn, open the main door of the enclosure (Figure 33.3).

To open the side door to access the oil pipes, lift the side door upwards and open it to the left.

NOTE: The main door must be open to allow the side door to open.

To close the main door, first close the side door then close the main door.

Press the interlock lever and firmly latch the main door locking mechanism onto the side door bar.

NOTE: Ensure that the interlock lever is correctly seated, and that the handle no longer moves before assuming the door is locked (Figure 33.5).

⚠

CAUTION: Both doors must be fully closed and the interlock lever must be engaged before pressurising the system.

Figure 33.1

Figure 33.2

Figure 33.3

Figure 33.4

5.2.2 Operation of the reaction vessel Bottom Outlet Valve (BOV)

Instructions for the operation of the BOV can be found in Batch Reactor Manual 1 – 2000255. However extra care should be taken with the Bottom Outlet Valve on a pressurized vessel and the following warnings should be read and fully understood before use.

CAUTION: Do not operate the Bottom Outlet Valve when the system is pressurised.

CAUTION: Do not over-tighten the Bottom Outlet Valve. The spring force inside the Bottom Outlet Valve is sufficient to hold the pressure inside the vessel. An approximate 1cm gap between the metal C-clip and the Bottom Outlet Valve knob ensures sufficient spring force is being applied to keep the vessel sealed. Tightening the Bottom Outlet Valve further introduces unwanted force that could cause the Bottom Outlet Valve or the vessel to break.

5.2.3 Pressurizing the Atlas Potassium 3 Bar Pressure System

For operations in this section, please refer to Figure 32.

To pressurize the Atlas Potassium 3 Bar Pressure System ensure that all gas connections are leak free.

1. Ensure that the safety enclosure door is fully closed and latched and additional safety screens are in place and secure.
2. Ensure that the input pressure from the gas supply is less than the vessel maximum allowable working pressure (MAWP).
3. Ensure that the gas inlet and outlet manual valves are closed.
4. Open the gas supply valve (regulator from a cylinder or valve from a hard-plumbed gas source).
5. Slowly open the gas inlet valve by rotating the black knurled knob anti-clockwise. Do not open fully and make sure to open slowly. Stop opening when the supplied gas flows into the pressure vessel.
6. The vessel will then be pressurized to the gas source pressure.
7. When the desired pressure is achieved, close the inlet valve by rotating the black knurled knob clockwise.
8. The system is now pressurized and ready for the experiment.

5.2.4 Purging the System

For operations in this section, please refer to Figure 32.

The Atlas Potassium 3 Bar Pressure Systems offer the ability to be able to purge the vessel with a stream of gas prior to pressurizing the system.

To purge the vessel:

1. Follow points 1-3 of Section 5.2.3 Pressurizing the Atlas Potassium 3 Bar Pressure System
2. Adjust the inlet and outlet valves to maintain a constant flow of gas through the vessel at the required purge pressure.
3. Close the inlet valve by rotating the black knob clockwise.
4. Vent the vessel by opening the gas outlet valve.
5. When all gas has been vented close the gas outlet valve.
6. Slowly open the gas inlet needle valve by rotating the black knob anti-clockwise (do not open fully) until the supplied gas flows into the pressure vessel.
7. The vessel is pressurized to the gas source pressure.
8. Close the inlet valve by rotating the black knob clockwise when the required pressure is reached.
9. Run the desired reaction ensuring all safety instructions have been adhered to.

CAUTION: Take great care if heating up the reaction vessel when the system is under pressure as the pressure will increase dramatically during a temperature rise. In some cases it may be preferable to pressurize the reaction vessel after the desired temperature has been achieved.

5.2.5 Depressurizing the System

For operations in this section, please refer to Figure 32.

After pressurizing the Atlas Potassium 3 Bar Pressure, it is important to fully depressurize the reactor.

1. Following the reaction and when the reactor has cooled back to room temperature, manually vent the pressure by slowly rotating the outlet valve black knurled knob anti-clockwise.
2. Ensure that the outlet gas is vented to a suitable exhaust in the laboratory.
3. Once the pressure is vented, close the outlet valve by rotating the black knurled knob on the manual release valve clockwise.
4. Check pressure gauge reads 0 Bar

5. It is now safe to open the door of the safety enclosure and the bottom outlet valve of the vessel can now be opened to remove the vessel contents (Please refer to Batch Reactor Manual 1 – Atlas Systems PN: 200025) for instructions on the use of the sprung bottom outlet valve).

5.2.6 Control of the Atlas Potassium 3 Bar Pressure System

Please refer to the Batch Reactor Manual 1 – Atlas Systems (PN: 2000255) for instructions on the manual use of the Atlas Potassium Pressure System Base.

Please refer to the Batch Reactor Software Manual 1 – Atlas and Reactor Master Software – 2000262 for instructions on the Atlas Potassium Pressure System within the Atlas 1.4 software.

6 Mass Flow Meters/Mass Flow Controllers

The Atlas Potassium 3 Bar Pressure System, Globe 3 Bar System and Atlas Sodium High Pressure System can be further upgraded by the addition of a Mass Flow Meters and/or Mass Flow Controllers.

Syrris supplies a range of Mass Flow Meters and Mass Flow Controllers from Aalborg. These are specifically tailored for the Syrris Systems and can be fully integrated into the Atlas and Reactor Master software platforms.

NOTE: It is important that the Aalborg Mass Flow Meter and Gas Flow Controllers are calibrated for the desired reaction gas.

The Mass Flow Meters (PN: 2200354 and 2200355) are ideal for applications which require accurate information on the amount of gas is flowed to the reactor. In particular, chemical reactions where reactive gases are used, the amount of gas uptake can be monitored.

The Mass Flow Controller (PN: 2200364) is ideal for applications where accurate dosing of gas to a reactor is required.

An example of a Mass Flow Meter supplied by Syrris is shown in Figure 34. All Mass Flow Meters and Controllers contain an inlet and an outlet fitting. Depending on the desired mode of operation, the units are inserted either immediately before or immediately after the reactor system.

Figure 34

Viewing data and programming on both the Mass Flow Controller and Mass Flow Meter is possible using the Atlas or Reactor Master Software (via an Atlas Port and the RS232 interface on the rear of the units). The Mass Flow Meter has an LCD display where data can be viewed and configuration options are available via keypad on the unit.

For further information on the configuration of Mass Flow Meters and/or Mass Flow Controllers in a full gaseous pathway please see section 8 Modes of Operation.

All Mass Flow Meters and Mass Flow Controllers are supplied with a manual which gives full information on the set-up, configuration and operation of the units. Please refer to the manual supplied with the Mass Flow Controller or Mass Flow Meter prior to operation.

Mass Flow Meters and Mass Flow Controllers are classed as 3rd party devices and require an Atlas Port (PN: 2101020) to connect to the Atlas and Reactor Master software. Please see Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (PN: 2000262) for information on automating the units.

7 3 Bar Autosampling

The 3 Bar Dip Tube for Autosampling (PN: 2200373) is designed specifically to aspirate up to 6 samples (when using 6 port valves) from a reactor pressurised up to 3 bar at user defined intervals, when used with the Autosampler Module and Atlas Syringe Pump.

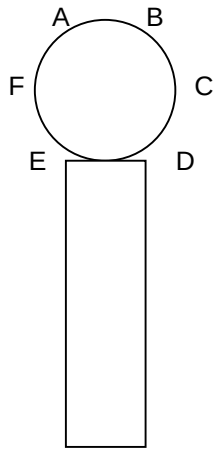
The Atlas Syringe Pumps (regular up to 20ml/min or XL up to 200ml/min) are versatile volumetric dosing and/or sampling systems. They allow 2 independent refilling flows, one continuous flow, reaction sampling, intelligent control of pH and temperature dependant dosing. An Atlas Syringe Pump or Atlas Syringe Pump XL consists of two syringes and multi-port valves and is controlled from the front panel, by an Atlas Base Unit, Reactor Master or the Atlas or Reactor Master PC software.

Information on how to use the Atlas Syringe Pump can be found in can be found in the Atlas Syringe Pump Manual (PN: 2000257).

The Autosampler Module, which can be mounted on the extra pair of Support Rods provided, holds 6 x 2ml HPLC vials and is located, in a fluidic sense, between the Atlas Syringe Pump and the 3 Bar Dip Tube for Autosampling (PN: 2200373). The Atlas Syringe pumps can sequentially aspirate samples from the reactor into the vials (which can contain a chemical quench if required).

The 3 Bar Dip Tube for Autosampling (PN: 2200373) consists of 6 PTFE pipes held in a chemically resistant (PTFE and glass) sheath, which can be located in a Jacketed Vessel Lid port using the probe holder included. Each of the PTFE pipes is connected to the Autosampler Module during use.

7.1 Fluidic setup



Port	Device connected to port
A	Reactor (3 Bar Dip Tube for Autosampling)
B	Waste Atmospheric pressure
C	Wash solvent or Inert gas (e.g. N_2) *Gas pressure must be the same as the reaction pressure!*
D	Vial 1 (vertical input of Autosampler)
E	Vial 2 (vertical input of Autosampler)
F	Vial 3 (vertical input of Autosampler)

Figure 34.1

The diagram above shows an example of the ports found on the Atlas syringe pump when a 6 port valve is connected. For more information about the ports, and making fluidic connections on the Atlas Syringe Pump, please

see the Atlas Syringe Pump Manual (PN: 2000247) Connect one output of the autosampler probe to Atlas pump channel 1 and the other output to Atlas pump channel 2.

Tubing information

NOTE: The tubing lengths and diameters are important and can be set on the Atlas Syringe Pump. Below are the default and recommended values.

Output volume of the tube from the valve to the autosample hardware default setting = 1.2m long tube of 1mm ID/1.6mmOD = 785µl

Volume of pipe from far end of 3 Bar Dip Tube for Autosampling to the Atlas Syringe Pump valve default setting = 1m long tube of 0.8mm ID/1.6mmOD = 395µl

7.2 3 Bar Dip Tube for Autosampling accuracy and limits

The quoted accuracy of the 3 Bar Dip Tube for Autosampling is +/- 50µl. This is applicable from atmospheric pressure all the way up to the operating pressure of 2.5 bar.

Minimum sample size: Based on the accuracy, it would be unwise to take a sample volume less than 200µl.

Maximum sample size: The vials installed in the autosampling manifold are 2ml vials, so this is the maximum sample that can be taken.

NOTE: accuracy data collected using water.

7.3 Setting the Atlas syringe pump to Autosample

Figure 35

7.3.1 Menu Location in Atlas Syringe Pump

For more information about how to use the LCD display on the Atlas Syringe Pump please see 2000257 - Atlas Syringe Pump Manual -print.pdf.

Starting in the Dual Dosing Mode, select the pump to be used. (Figure 35)

In the next sub menu select option 3 OTHER COMMANDS. (Figure 36)

Figure 36

The new 3 BAR AUTOSAMPLE option will be inserted as option 4, below the normal AUTOSAMPLE option. (Figure 37)

Selecting **3 BAR Autosample** if the valve on the syringe has <= 3 ports should result in an error message **THIS NEEDS VALVE > 3**, which displays for 5 seconds, or until the user presses either the knob or the button and then returns to this menu.

Figure 37

7.3.2 Sub menu of “3 BAR AUTOSAMPLE”

1) START
2) SAMPLES

3) SAMPLE
4) WASH CYCLES
5) WASH
6) 1ST DELAY
7) DELAY
8) FILL
9) EMPTY
10) OUT VOL
11) RETURN VOL

7.3.3 Definition of Sub menu parameters

Start

Clicking on this starts the autosample operation and moves the display back to the top screen in Dual Dosing Mode.

Samples

Defines number of samples to be collected

Minimum = 1

Maximum = Valve Size - 3

Default = 1

Sample volume. Minimum 0.1 µl, Maximum Syringe volume. Default = 100µl

Wash cycles. Minimum 0, Maximum 9. Default=1

Wash volume

Defines the volume to be aspirated into the syringe and dispensed to waste

Minimum = smallest of Return Volume or Syringe volume

Maximum = Syringe volume

Default = 200 µl

First delay. Minimum 0. Maximum 999 mins. Default = 2 mins

Delay. Minimum Time taken for one sample operation with current parameters, see below. Maximum 999 mins. Default = 2 mins

FILL. Fill rate. Min is min flow rate for syringe volume, Max is max flow rate for syringe volume. Default is the current default fill rate for syringe.

EMPTY. Empty rate (or dispense but that's too long a word!). Min is min flow rate for syringe volume, Max is max flow rate for syringe volume. Default is the current default fill rate for syringe.

OUT VOL. Output volume of the tube from the valve to the autosample hardware.

RTN VOL. Volume of pipe from far end of Autosampler Probe to the Atlas Syringe Pump valve.

The minimum value allowed for Delay needs to be calculated from the existing parameters such that. (Delay needs to be at least as long as the time taken to run one complete sample).

Minimum Delay Time = Wash Time + Sample Time + Wash Tube Time + Return Time

Wash Time = Wash cycles * (Wash volume / Fill Rate + Wash volume / Empty Rate + valve switch time * 2 + Buffer Time)

Effective Sample Vol = Sample volume + Output Tube Volume

Sample Time =Valve Switch Time + Effective Sample Vol / Fill Rate + Valve Switch Time + Depressurize Time + Valve Switch Time + Effective Sample Vol / Empty Rate + Buffer Time

Effective Wash Tube Volume = Wash Tube Vol * 1.1

Wash Tube Time = Valve Switch Time + Effective Wash Tube Volume * (1 / Fill Rate + 1 / Empty Rate) + Buffer Time

Return Time = Return volume / Fill Rate + Valve Switch Time + Return volume / Empty Rate + Buffer Time

Valve Switch Time = 1 second

Buffer Time = 1 second (Extra time due to communications or other delays not accounted for)

Out Vol default setting = 1.2m long tube of 1mm ID = 785µl

Return vol default setting = 1m long tube of 0.8mm ID = 395µl

7.3.4 Definition of tasks after “Start” is selected

Step	Task	Details
1	Wait	Wait for the time defined in “First Delay”
2	Wash line A and syringe	Subroutine 1
3	Take sample and dispense to Vial X	Subroutine 2
4	Clean distribute tube X and dispense to reactor	Subroutine 4

5	Return contents of line A to reactor	Subroutine 3
6	Wait	Wait for the time defined in “Delay”
7	Repeat steps 2 to 5 for each sample to be taken	Repeat such that the total number of cycles equals “Samples”, with each cycle dispensing in subsequent vials (1, then 2, then 3)

Subroutine 1 (Wash line A and syringe)

Step	Task	Details
1	Aspirate from Port A	Aspirate “Wash volume”
2	Dispense to Port B	Dispense “Wash volume”
3	Repeat steps 1 and 2	Repeat “Wash cycles” times

Subroutine 2 (Take sample and dispense to vial)

Step	Task	Details
1	Aspirate from Port A	Aspirate “Sample volume” + Output Tube Volume
2	De-pressurise at Port B	Stop at Port B for 1 second to de pressurise the syringe
3	Dispense to Vial X (Port D,E or F)	Dispense “Sample volume” + Output Tube Volume to Vial

Subroutine 3 (Return contents of line A to reactor)

Step	Task	Details
1	Aspirate from Port C	Aspirate “Return volume”
2	Dispense to Port A	Dispense “Return volume”

Subroutine 4 (Clean distribute tube X and dispense to waste)

Step	Task	Details
1	Aspirate vol of distribute tube X	Aspirate vol of distribute tube X + 10% to ensure it’s all been aspirated
2	Dispense to port B	Dispense entire contents of syringe back into port A

7.4 Implementation in Atlas PC Software

There is an action category called **atlas 3 Bar Autosample**

This is available in the Recipe Action outlook bar selection when an Atlas Syringe pump is present on the hardware screen.

Dragging the action onto a recipe will generate an editor.

Clicking ok, will cause the editor to validate the values inputted.

For more information about using the Atlas Software please see Batch Reactor Software Manual 1 – Atlas and Reactor Master Software (2000262)

Note: Important information about the syringe pump will **not** be available to the PC. This means the validation for things like volume and delay times cannot be done.
The consequence of this is that the pump may reject the autosample command once that step is reached in the recipe, causing the recipe to fail at run time or simply skip the autosample step.

7.5 Troubleshooting

The Atlas software skips the 3 bar autosample step

This is commonly caused by not having a long enough delay time for all the steps in the sub routine to complete. Remember the minimum value allowed for Delay needs to be calculated from the existing parameters (Delay needs to be at least as long as the time taken to run one complete sample).

Minimum Delay Time = Wash Time + Sample Time + Wash Tube Time + Return Time

8 Modes of Operation

There are several modes of operation available for the Atlas Potassium 3 Bar Pressure system and the Globe 3 Bar system. These modes of operation depend on the desired application and the hardware that is used. The available hardware used in addition to the Syrris Pressure systems to allow the modes of operation described below are:

- 3 Bar Gas Pressure Control Module (PN: 2200497)
- Gas Selection Module (PN: 2200375)
- Mass Flow Meters/Mass Flow Controller (see Batch Chemistry Reactors Catalogue for part number details and options)
- Automated control using Syrris PC software (see Batch Chemistry Reactors Catalogue for part number details and options)

Each mode of operation will consist of a different fluidic set up. For any assistance in configuring the hardware for a specific application, please contact Syrris at support@syrris.com

8.1 Manual Pressure Control

In standard configuration, the Atlas and Globe Pressure systems can be used with manual valves to control and pressurize the reactor. Figure 38 shows a schematic of the hardware set up.

In this mode, the manual and outlet valves begin in the closed position. The external gas supply must be equal to or less than the maximum pressure rating of the reactor.

To pressurise the system:

1. Check the input gas pressure is lower than the MAWP of the vessel.
2. Turn on the external gas supply.
3. Open the manual Inlet needle valve. This must be done slowly.
4. Close the Inlet valve.
5. The system should now be pressurised and assuming that there are no leaks, the system will maintain the desired pressure.

Pressure is displayed via an analogue gauge and is not automatically data logged. A Pressure Sensor (PN: 2200370) and Pressure Node (PN: 2200369) can be added to automatically log and display pressure values.

To vent the reactor after the reaction, slowly open the Outlet manual needle valve and ensure the gas is vented to a suitable exhaust.

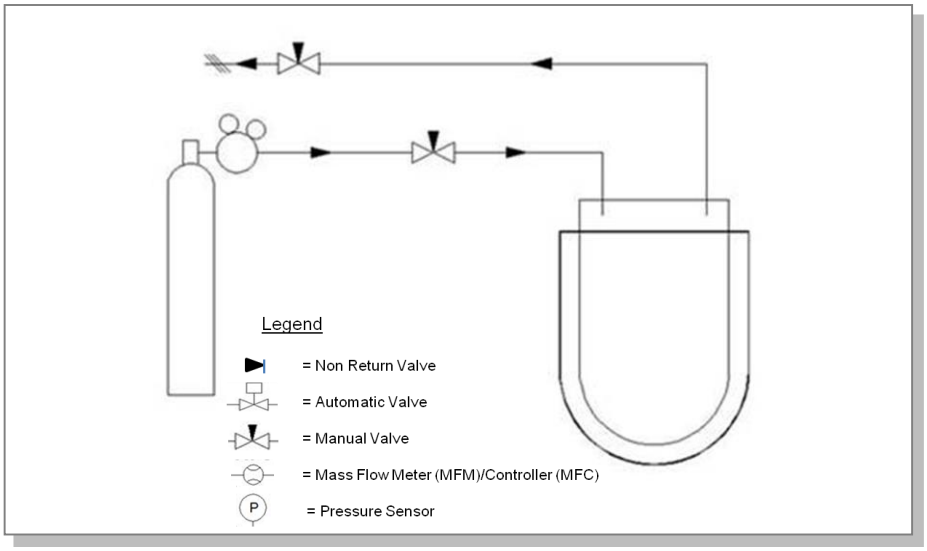


Figure 38

8.2 Automatically Maintain a Pressure

Adding a 3 Bar Gas Pressure Control Module (PN: 2200497) to the system allows the reaction pressure to be automatically controlled during an experiment.

The Gas Pressure Control Module is connected as shown in**Error! Reference source not found.**. For more information on the set up and operation of this module, please read Batch Reactor Manual 8 – Gas Pressure Control Module (PN: 2000285).

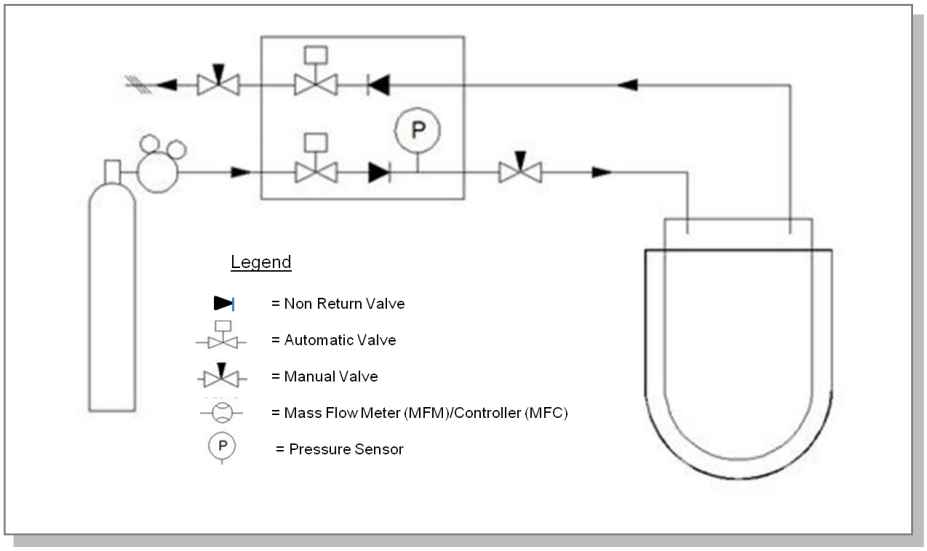


Figure 39

The Actual and Set pressures are displayed on the module and the system can be controlled either manually using the control knob on the unit or automatically using the Atlas or Reactor Master Software. The system can be used to purge and vent the reactor in addition to automatic pressure control.

CAUTION: Care must be taken on set up to ensure that there is correct flow control on the inlet and outlet of the reactor. This is achieved by adjusting the Gas Inlet and Outlet Valves and restricting both flows. If the Gas Outlet Valve has a greater restriction than the Gas Inlet Valve, there is a high risk of pressure rise within the reactor.

8.3 Automatic Pressure Control with Mass Flow Monitoring

Adding a Mass Flow Meter to the configuration described in section 8.2 Automatically Maintain a Pressure, allows active pressure control of the reactor with a measurement of the gas used in the reaction. This is a very common configuration and is used extensively for gas uptake/ gas insertion reactions (such as Hydrogenations, Carbonylations, etc.).

The schematic of the configuration is shown in Figure 39. The Mass Flow Meter is situated after the Gas Pressure Control Module and before the inlet to the reactor. A Mass Flow Meter can be used without the Gas Pressure Control Module to perform similar gas uptake measurements if active pressure control is not required.

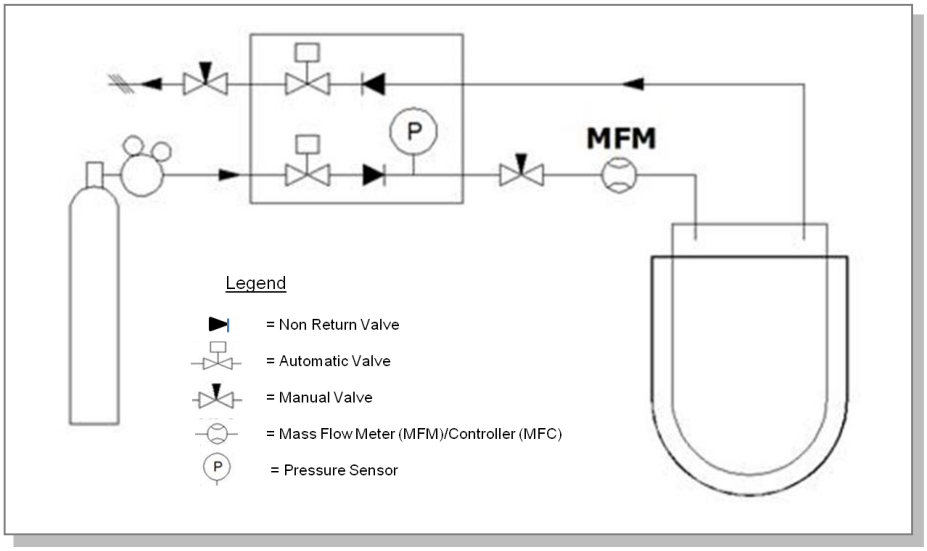


Figure 40

A typical method for this operation is described below:

CAUTION: Input pressure to the Gas Pressure Control Module must be less than the Maximum Allowable Working Pressure (MAWP) of the reaction vessel.

1. Charge the reactor with reagents (usually performed with an open vessel or through large ports as reagents can be solid catalyst and / or liquid reagents)
2. Seal the vessel.
3. Pressurize the system with inert gas to ensure there are no leaks
4. Purge the system with inert gas to remove all air (alternatively perform a vacuum and degas of the reactor with inert gas)
5. Begin pressure control using the Gas Pressure Control Module. This introduces the reactive gas.
    a. For gas insertion reactions (such as Hydrogenation), the gas that is introduced will react with the reagents in the reactor
6. The Mass Flow Meter will monitor the amount of gas that is being used in the reaction.
7. The reaction is complete when no more flow of gas is seen on the Mass Flow Meter. This can be viewed on the display where no further increase will be observed.
8. The system can then be vented using the Gas Pressure Control Module.
9. Addition of a Gas Selection Module in this configuration allows the automatic introduction of an inert gas after the reaction.

8.4 Mass Flow Control

A number of applications require accurate dosing of a gas to a reactor. This can be achieved by adding a Mass Flow Controller to the system configuration. This is depicted in Figure 41.

The Mass Flow Controller is situated at the inlet of the reactor after the Gas Pressure Control Module. A Mass Flow Controller may be used with or without a Gas Pressure Control Module. When a Gas Pressure Control Module is present, the system can be set so that it vents when a certain pressure is achieved. This is particularly important as the Mass Flow Controller will constantly add gas to the reactor. If a Gas Pressure Control Module is not present, care must be taken so that there is no risk of a pressure build up. A Mass Flow Controller is often used when a controlled purge is required under atmospheric pressure conditions.

CAUTION: Input pressure to the system must be less than the Maximum Allowable Working Pressure (MAWP) of the reaction vessel.

CAUTION: Mass Flow Controller flow rate must be suitable to the reactor system. A high flow rate may result in a large pressure increase.

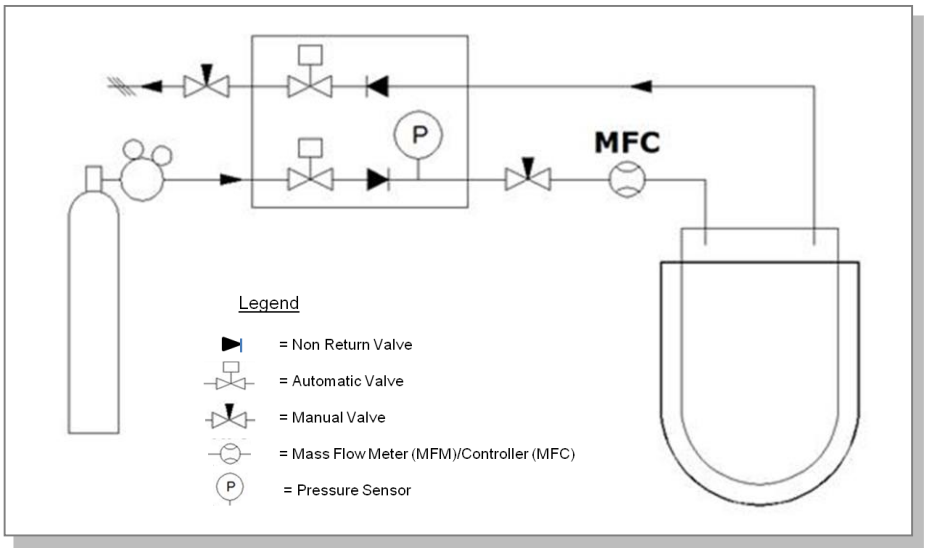


Figure 41

8.5 Off-Gas Monitoring

In certain applications, a reaction can generate an off-gas during the process which may then be measured. This can be achieved by adding a Mass Flow Meter or an alternative off-gas monitoring system at the outlet of the reactor. For advice on configuring a system for off-gas monitoring, please contact Syrris at support@syrris.com.

8.6 Multiple Gas Selection

All of the above modes of operation can be upgraded by the use of a Gas Selection Module (2200375) which enables up to three separate gases to be plumbed into the gaseous pathway simultaneously, and can either be controlled manually, or via the Atlas and Reactor Master software.

The Gas Selection Module is plumbed into the gaseous pathway before the Pressure Control Module. Please see the Batch Reactor Manual 9 – Gas Selection Module (PN: 2000286) for more information on the configuration and operation of this module.

For more information on setting up a particular experimental configuration or for advice on how to apply the Syrris range of hardware to achieve a specific application, please contact Syrris at support@syrris.com.

9 Hardware Maintenance

9.1 Standard Part Maintenance

Item	Frequency	Action
All glass vessels, lids, condensers etc.	Before each reaction	Check for chips and cracks If present, replace the item before use
All items	Every 3 months	Clean system as described above in the “cleaning” section
All reaction/reagent wetted parts	After each reaction	Clean thoroughly and dry
Bottom outlet valve	Every 3 months or if: a) the valve does not move freely b) the bottom outlet valve is leaking	1) Totally remove the bottom outlet valve by fully unscrewing then pulling downwards 2) Clean with suitable solvent s (e.g. water then acetone). 3) If tolerated, apply a thin film of vacuum grease to the outside of the o-rings 4) Re-insert the bottom outlet valve
Bottom outlet valve	Annually or after 2000 hours use	1) Totally remove the bottom outlet valve by fully unscrewing then pulling downwards 2) Clean with suitable solvent s (e.g. water then acetone) 3) Replace the black o-rings (Part numbers 1802152 and 1802042) 4) Re-insert the bottom outlet valve
Circulator	Please refer to the user manual of the circulator	Please refer to the user manual of the circulator
Scorpion Overhead Stirrer Drive Head	Every 3 years or 6000 hours use	1) Replace Scorpion Overhead Stirrer Drive Head (Without Pole), part number 2101120
PTFE Stirrer	Every 6 months or 1000 hours use	1) Check PTFE stirrer shaft for signs of damage 2) Replace if damaged to avoid stirrer slipping
Safety enclosure	Every 3 months	Check to make sure the door cannot be pulled open without operating the thumb operated interlock. Check for any obvious signs of corrosion from Chemicals that may affect the enclosures performance. If any is found contact Syrris for advice.
Safety Enclosure screen	When required by user	1) From the inside of the enclosure, slide the screen out of the door by lifting it over the retaining lip on the long edge of the screen and sliding it out of its support rails. 2) Order a new screen from Syrris (Part number 2200714) and replace by doing the reverse of step 1

9.1.1 Atlas Potassium 3 Bar Pressure Relief Valve Test Protocol

The following procedure may be performed to test the relief pressure of the Pressure Relief Valve supplied with the system. This is tested by pressurising the valve and observing the flow at the exhaust of the relief valve. At the point at which flow escapes through the relief valve, the pressure value can be observed on the inlet.

Test Protocol Procedure:

1. Use a regulated supply of clean, dry non-hazardous gas that is able to supply up to 3.0bar of pressure.
2. Fill a beaker with approximately 500ml of water. This is used to observe bubbles from the relief valve exhaust.
3. Open the manual release (outlet) valve, and close the Inlet valve on the 3 Bar System Safety Manifold.
4. Disconnect the gas outlet from the Safety Manifold to the Pressure Vessel, and fit a Swagelok™ ¼ blanking plug (SS-400-P) to the outlet elbow.
5. Fit tubing (either silicone hose or ¼" OD polymer tube) to the outlet port of the pressure relief valve.
6. Place the other end of the tubing under the water in the beaker.
7. Slowly open the Inlet valve until gas is heard flowing from the manual release valve.
8. Close the manual release valve slowly whilst observing the pressure. Stop if it seems to be rising rapidly, and ensure that the supply is less than the maximum pressure rating of the vessel (MAWP).
9. When the manual release valve has been closed there should be no audible leaks, and no bubbles from the pressure relief valve outlet port.
10. Slowly increase the inlet pressure to 2.5bar, observing the pressure gauge.
11. Constantly check the outlet tube in the water for bubbles.
12. Increase the pressure in 0.1bar increments, with a 30 second delay between each increase to check if a bubble has started to form.
13. Note the pressure at which a bubble has started to form. This pressure is the relief pressure.

NOTE: The relief pressure is factory set to 2.75 Bar (+/- 0.05 Bar)

NOTE: Do not under any circumstances attempt to pressurise the system above 2.75 Bar!

NOTE: If the valve fails to provide bubbles within the specified limits, contact Syrris or an approved representative immediately.

9.1.2 Atlas Potassium 3 Bar Pressure System, Globe 3 Bar Pressure System and Atlas Sodium High Pressure System Rupture (Burst) Disc Replacement Protocol

It may be necessary to replace a Rupture (Burst) Disc. All replacement Rupture Discs are supplied with detailed replacement instructions.

9.1.3 Safe protocol for finding leaks in pressurised system

If it is suspected that the system has a leak somewhere then the leak needs to be found and corrective action taken. This is a guide on how to do this safely.

Leak finding Protocol Procedure:

1. Ensure all fittings are tight and the system is fully assembled.
2. Make sure the Syrris Safety Enclosure is closed with the interlock lever fully engaged as explained in section 5.2.1 and full PPE is worn.
3. Fill the pressure vessel as much as possible with water so that there is a very small air gap between the fill level and the underside of the lid.
4. Slowly increase the pressure of the system up to 1 bar pressure
5. Use Snoop™ soap solution to detect leaks. A leak is present if the solution bubbles or foams. Check the following areas.
    a. All connections on the vessel lid including probe holders.
    b. All connections on the safety manifold.
    c. All connections on Gas Selection module and Gas Pressure Control module.
6. If no leaks can be found then it could be the gas supply external to the Syrris system that is leaking

NOTE: Snoop™ solution can stain anodised aluminium parts. Be sure to clean it off after completing leak finding protocol.

10 Cleaning Systems

Material	Description	Example(s)	Cleaning
Epoxy powder coating	White/cream painted surfaces of Syrris parts	Main covering of Base Unit, Atlas Syringe Pump	Surfaces may be cleaned with a damp cloth with mild detergent In extreme cases, a cloth dampened with iso-propanol may be used
Metal surfaces	Stainless steel, regular anodised aluminium, red anodised aluminium, gold	Jacketed Vessel Clamp, Scorpion Overhead Stirrer Pole, Oil Drain Unit	Surfaces may be cleaned with a damp cloth with mild detergent In extreme cases, a cloth dampened with iso-propanol or acetone may be used.
PTFE surfaces	Opaque white polymer	RTD probe, stirrer, “flexible” section of Scorpion Overhead Stirrer Pole	
PEEK surfaces	Beige coloured polymer	Node and Scorpion Overhead Stirrer connections on the Base Unit	
Polypropylene surfaces	Partially translucent white/cream polymer	Dry Bath safety shroud, Orbit Reflux Module	
Regular power and communications connections (excludes scorpion overhead stirrer connections and large [30mm] node connections and as these are PEEK/Gold)	Various	USB ports, power sockets, cables, connections for the hotplate and magnetic stirrer	Do not clean Be sure to avoid when cleaning surfaces close to these connections Do not allow ingress of moisture or solvent vapour
All other surfaces not described above	Various	Exterior of oil pipes, other manufacturer’s equipment e.g. circulators, other surfaces not described above	Damp cloth with mild detergent

11 Contacting Syrris

For technical support, please email support@syrris.com

For all other enquiries please email info@syrris.com

For more detailed contact details, please see the back cover of this document.

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12 Appendix

12.1 EC Declaration of Conformity

We Syrris Ltd
of 27 Jarman Way, Royston, Herts, SG8 5HW

in accordance with the following Directive(s):

2004/108/EC	The Electromagnetic Compatibility Directive
2006/42/EC	The Machinery Directive

hereby declare that:

Equipment: Atlas reactor system consisting of:

Part Number	Description
2101000	Atlas Base Unit
2200072	Atlas Syringe Pump
2200100	Atlas Syringe Pump - without Valves or Syringes
2200376	Atlas Syringe Pump XL
2200316	Atlas Syringe Pump XL - without Valves or Syringes

and associated accessories are in conformity with the applicable requirements of the following documents:

Ref. No.	Title	Edition/date
EN ISO 12100	Safety of machinery — General principles for design — Risk assessment and risk reduction	2010
EN 61010-1:2010	Safety requirements for electrical equipment for measurement, control, and laboratory use. General requirements	2010
EN 61000-6-1	Electromagnetic compatibility (EMC) - Part 6-1: Generic standards - Immunity for residential, commercial and light-industrial environments	2007
EN 61000-6-3	Electromagnetic compatibility (EMC) - Part 6-3: Generic standards - Emission standard for residential, commercial and light-industrial environments	2007

I hereby declare that the equipment named above has been designed to comply with the relevant sections of the above referenced specifications. The unit complies with all applicable essential requirements of the Directives.

Signed by:

Name: Mike Hawes

CE 12

Position: CEO

Done at Syrris Ltd

27 Jarman Way,
Royston, Herts,
SG85HW, UK

On 30th August 2012

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12.2 CERTIFICATE OF DECONTAMINATION

This document must be completed in full and signed by the Customer (a) before transporting, by any means, a product to Syrris for repair or service, and (b) prior to any on-site service to be performed by Syrris Ltd.
A completed copy of this Certificate is required to be attached to the instrument / part itself AND to the outside of the packing container when transporting to Syrris Ltd or Syrris Inc.

- Please ensure all wetted parts have been thoroughly washed with methanol, ethanol, Isopropyl alcohol (IPA) or acetone.
- Please ensure any glassware has been thoroughly washed with methanol, ethanol, Isopropyl alcohol (IPA) or acetone.
- Please ensure any items that are being sent to Syrris is suitably packaged and well protected.
- Please write a brief description for the reason of this/these items are being sent to Syrris.

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- Please write down what substances the item(s) have been in contact with most recently.

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Sign Name	
Print Name	
Date	

Company Name	
Address 1	
Address 2	
Address 3	
Town/City	
Post Code	
Country	

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For the quickest response for all technical enquiries please email
support@syrris.com

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